IS 5042 (1976): 1-aminoanthraquinone [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

BLANK PAGE

IS : 5042 - 1976

Indian Standard

SPECIFICATION FOR 1-AMINOANTHRAQUINONE

(First Revision)

UDC 667·282·14 :547·673·5

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Price Rs 5·00

February 1977

AMENDMENT NO. 1 SEPTEMBER 1986

TO

IS: 5042-1976 1-AMINOANTHRAQUINONE

(First Revision)

[*Page 4, Table 1, Sl No. (i), col 3*] - Substitute '96.0' *for* '95.0'.

(PCDC 11)

Reprography Unit, ISI, New Delhi, India

Indian Standard

SPECIFICATION FOR 1-AMINOANTHRAQUINONE

(First Revision)

Dye Intermediates Sectional Committee, CDC 46

Chairman	*Representing*
SHRI RAJENDRA SHANKAR	Indian Dyestuff Industries Ltd, Bombay
Members	
SHRI M. S. TAWAKLAY (*Alternate* to Shri Rajendra Shankar)	
SHRI P. B. BHATTACHARJEE	Luxmi Narayan Dyestuff & Chemical Works Pvt Ltd, Calcutta
SHRI B. M. BRAHME	Atic Industries Ltd, Bulsar
DR M. M. DESHPANDE SHRI N. G. AMIN (*Alternate*)	Suhrid Geigy Ltd, Vadodara
SHRI JASTINDER SINGH	Directorate General of Technical Development, New Delhi
SHRI G. A. KULKARNI SHRI S. V. DESAI (*Alternate*)	Amar Dye-Chem Ltd, Bombay
SHRI S. M. MEHTA SHRI M. V. DESAI (*Alternate*)	Atul Products Ltd, Bulsar
SHRI Y. R. MEHTA SHRI N. C. BAKSHI (*Alternate*)	I.C.I. (India) Pvt Ltd, Calcutta
DR P. N. PANDIT SHRI H. K. VENKATARAMAIAH (*Alternate*)	Hindustan Organic Chemicals Ltd, Rasayani (Maharashtra)
DR R. J. RATHI SHRI K. L. RATHI (*Alternate*)	Sudarshan Chemical Industries Pvt Ltd, Pune
SHRI SANTOKH SINGH SHRI KULWANT SINGH (*Alternate*)	National Chemical Industries Pvt Ltd, New Delhi
DR H. A. SHAH SHRI G. G. SUTAONE (*Alternate*)	Development Commissioner, Small Scale Industries, New Delhi
SHRI S. J. SHAH SHRI M. B. MEHTA (*Alternate*)	Indian Chemical Manufacturers' Association, Calcutta

(*Continued on page* 2)

(*Continued from page* 1)

Members	*Representing*
DR D. R. SRIDHAR DR R. N. DHAR (*Alternate*)	Indian Drugs & Pharmaceuticals Ltd, New Delhi
DR P. V. SUBRAMANIAM	Colour-Chem Ltd, Bombay
SHRI MUKUND TURAKHIA SHRI ANIL MEHTA (*Alternate*)	The Dyestuffs Manufacturers Association of India, Bombay
SHRI V. G. UPADHYE	Synthofine Chemicals of India Pvt Ltd, Bombay
DR G. M. SAXENA, Director (Chem)	Director General, ISI (*Ex-officio Member*)

Secretary

SHRI NARESH K. SHARMA
Deputy Director (Chem), ISI

Panel for Anthraquinones, CDC 46 : P 7

Convener	
SHRI RAJENDRA SHANKAR	Indian Dyestuff Industries Ltd, Bombay
Members	
SHRI M. S. TAWAKLAY (*Alternate* to Shri Rajendra Shankar)	
SHRI B. M. BRAHME	Atic Industries Ltd, Bulsar
SHRI G. A. KULKARNI SHRI V. K. MENON (*Alternate*)	Amar Dye-Chem Ltd, Bombay
DR P. V. SUBRAMANIAM	Colour-Chem Ltd, Bombay

Indian Standard

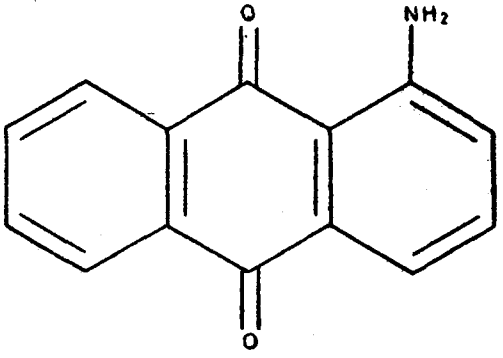
SPECIFICATION FOR 1-AMINOANTHRAQUINONE

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 25 August 1976, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.

0.2 1-Aminoanthraquinone ($C_{14}H_9O_2N$) is an important dye intermediate used in the manufacture of vat and disperse dyes. It has the following structural formula:



1-AMINOANTHRAQUINONE
(Molecular Mass 223·2)

0.3 This standard was first issued in 1969. In the present revision, the requirements for melting point and benzene insolubles along with their methods of test have been included.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 1-aminoanthraquinone.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of brownish-red crystalline powder and shall be free from visible impurities.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 1-AMINOANTHRAQUINONE

Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No. in Appendix A
(1)	(2)	(3)	(4)
i)	Assay, percent by mass (on dry basis), *Min*	95·0	A-2
ii)	Moisture content, percent by mass, *Max*	0·5	A-3
iii)	Sulphated ash, percent by mass (on dry basis), *Max*	1·0	A-4
iv)	Benzene insolubles, percent by mass (on dry basis), *Max*	2·0	A-5
v)	Melting point (on dry basis)	Within 3°C including the temperature 250°C	A-6

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1970*) lined with suitable polyethylene film, or as agreed to between the purchaser and the supplier. The containers shall be securely closed.

3.2 Marking — Each container shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Batch number; and

d) Tare, net mass and gross mass.

*Specification for steel drums (galvanized and ungalvanized) (*first revision*).

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in **3** of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Test for assay shall be conducted on each of the individual samples.

4.2.2 Tests for the remaining characteristics, namely moisture content, sulphated ash, benzene insolubles and melting point shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirement of assay if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of characteristics tested on the composite sample (*see* **4.2.2**), the test results for each of the characteristics shall satisfy the relevant requirement given in Table 1.

5. TEST METHODS

5.1 Tests shall be carried out according to the methods prescribed in Appendix A, as indicated in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1060†) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Methods of sampling and tests for dye intermediates.

†Specification for water, distilled quality (*revised*).

APPENDIX A

(*Table* 1 and *Clause* 5.1)

METHODS OF TEST FOR 1-AMINOANTHRAQUINONE

A-1. PREPARED SAMPLE

A-1.1 Dry the material at 105 ± 1°C to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this *prepared sample* for tests except for moisture determination.

A-2. ASSAY

A-2.0 Outline of the Method — Purity of the material is estimated by chromatographic method. 1-Aminoanthraquinone is separated chromatographically and determined quantitatively by spectrophotometer.

A-2.1 Apparatus

A-2.1.1 *Chromatographic Column* — a glass tube, 40 cm long, about 1·5 cm in internal diameter, joined with a 50-ml thistle funnel at the upper end and fitted with a stop-cock at the lower end.

A-2.1.1.1 Set up the column vertically. Place a cotton-wool plug in the tube and press to the bottom by means of a glass rod flattened at the end. Place a disc of filter paper cut to the approximate internal diameter of chromatographic tube on top of the cotton-wool.

A-2.1.1.2 Prepare a slurry of about 15 g of alumina in toluene and pour it into the tube. Wash down the sides of the tube and pack the column by light tapping. Place first a disc of filter paper and then a cotton wool plug on the top surface of alumina column.

A-2.1.1.3 Always keep enough solvent in the column so that it remains wet and that at least 2 cm of solvent layer always persists over the top of alumina. On no account allow the alumina to run dry. In the event of this happening, reslurry the alumina and repack.

A-2.1.2 *Spectrophotometer* — The readings of optical density or percentage transmittance are taken at wavelength of maximum absorption. This is obtained by plotting absorption against wavelength on a graph. The instrument shall be checked for accuracy from time to time.

A-2.2 Reagents

A-2.2.1 *Toluene* — dried for 24 hours over calcium chloride, filtered and distilled. Collect the portion boiling between 110 and 112°C for use in the test. The optical density shall not exceed 0·005 at 460 nm.

A-2.2.2 *Alumina* — neutral chromatographic grade. To get a material of desired activity treat it as in **A-2.2.2.1**.

A-2.2.2.1 Prepare a solution containing 20 to 30 mg of pure 1-aminoanthraquinone in 100 ml of toluene. Set up a chromatographic column as in **A-2.1.1** and chromatograph the solution. A compact main band shall be obtained, it shall be well separated from any earlier or later bands, and it shall be completely washed through the column with the addition of not less than 50 ml and not more than 150 ml of the eluent. If the chromatogram is unsatisfactory in any of these three respects and the alumina is insufficiently active, reject it. If the alumina is too active, treat it as follows:

> Spread out the alumina evenly on a stainless steel tray in a thin layer. Add the necessary amount of water slowly from a burette covering the whole of the tray as evenly as possible. Mix the alumina by hand initially and then transfer to a Kilner jar and mix by rolling mechanically for at least 2 hours. Then retest for activity. If still too active, repeat the procedure adding more water in increments of 0·5 percent.

A-2.2.3 1-*Aminoanthraquinone* — chromatographically pure.

A-2.2.3.1 Crystallize technical grade 1-aminoanthraquinone from toluene using animal charcoal for absorption of impurities. Repeat this procedure of crystallization three or four times. The crystals obtained after final crystallization are almost pure. To make chromatographically pure material, prepare a column of 350 g of neutral alumina in toluene as in **A-2.1.1**. Dissolve 1 g of crystallized 1-aminoanthraquinone in minimum quantity of toluene and transfer it gradually to the column. Allow the solution to pass through the column and elute further by toluene. Reject the coloured bands of impurities and collect the reddish-orange band of 1-aminoanthraquinone. To get a sizeable quantity of the pure substance, repeat this procedure a few times. Concentrate the combined eluates by distillation under reduced pressure. Filter the concentrate and allow the filtrate to crystallize. Filter the pure crystals and dry them under vacuum at 100 to 105°C.

A-2.2.3.2 To check the purity of the material, dissolve about 60 to 70 mg, accurately weighed, in 250 ml of toluene in a volumetric flask as in **A-2.3.1**. Pass 10 ml of this solution through the alumina column in the usual manner, and dilute to 250 ml in a volumetric flask. Pipette out 10 ml of the original solution into another 250-ml volumetric flask and dilute it to the mark with the solvent. Find out the optical densities of these solutions at 460 nm. The difference between the two readings should not be more than 0·003. If the difference is more, repeat the purification procedure.

A-2.3 Procedure

A-2.3.1 Weigh accurately about 60 mg of the *prepared sample* (**A-1.1**) in a 500-ml dry conical flask. Add about 150 ml of toluene and reflux for 15 to 25 minutes. Cool and transfer the solution to a 250-ml volumetric flask. Wash the conical flask well with toluene and transfer the washings to the flask. Dilute the solution to the mark with toluene.

A-2.3.2 Drain the excess solvent from the chromatographic column so that the top remains just wet. Transfer with the help of a pipette 10 ml of the sample solution accurately and slowly to the top of the column. Add the solution to the column dropwise and at the same time drain the solvent from the column at the same rate without allowing the top of the column to go dry. Fill the upper part of the tube with solvent and allow the chromatogram to develop, keeping a good head of solvent above the alumina throughout. Reject the coloured bands preceding the main orange coloured band of 1-aminoanthraquinone.

A-2.3.3 Start collecting fractions in a 250-ml volumetric flask when the main band is about 1·5 cm from the lower tip of the tube. When the entire 1-aminoanthraquinone band is eluted, remove the volumetric flask and dilute the contents to the mark with toluene. This is the test solution.

A-2.3.4 Adjust the wavelength of maximum absorption to the pre-determined value and then adjust the instrument in such a manner that the transmittance through the blank becomes 100 percent after inserting the cell with blank solution. Replace the cell with the solution of the sample and read the optical density or percentage transmittance. In the case of the latter refer to the standard conversion tables and find out the corresponding optical density.

A-2.3.5 Dissolve about 65 mg of accurately weighed pure substance into a 250-ml volumetric flask following the procedure for making the solution as prescribed in **A-2.3.1**. Pipette out 10 ml of this solution into another 250-ml volumetric flask and dilute it to the mark using toluene. Find out the optical density of this solution.

A-2.4 Calculation

$$\text{Assay, percent by mass} = \frac{A}{M} \times \frac{M_1}{A_1} \times 100$$

where

A = optical density of the material,

M = mass in g of the material taken for the test,

M_1 = mass in g of pure 1-aminoanthraquinone, and

A_1 = optical density of pure 1-aminoanthraquinone.

A-3. DETERMINATION OF MOISTURE CONTENT

A-3.1 Determine the moisture content of the material as prescribed in **9.3** of IS : 5299-1969*.

A-4. DETERMINATION OF SULPHATED ASH

A-4.1 Determine the sulphated ash of the *prepared sample* (**A-1.1**) as prescribed in **11.2** of IS : 5299-1969*.

A-5. DETERMINATION OF BENZENE INSOLUBLES

A-5.1 Apparatus

A-5.1.1 *Conical Flask* — 500 ml capacity, with ground socket joint.

A-5.1.2 *Liebig Condenser* — with ground glass joints.

A-5.1.3 *Calcium Chloride Guard Tube* — with ground glass joint.

A-5.1.4 *Sintered Glass Crucible* — G No. 4.

A-5.1.5 *Air-Oven* — preferably electrically heated with temperature control device.

A-5.2 Reagent

A-5.2.1 *Benzene* — dry, distilled.

A-5.3 Procedure — Weigh accurately about 1·5 g of the *prepared sample* (**A-1.1**) into the conical flask and add to it 300 ml of benzene. Heat under reflux condenser for 30 minutes. Cool slightly and filter the hot solution through a previously weighed crucible. Wash the flask and the crucible with hot benzene till the washings are colourless. Place the crucible in the air-oven for 1 hour at 105 ± 1°C. Remove the crucible, cool in a desiccator and weigh. Repeat the operations of heating, cooling and weighing till constant mass is obtained.

A-5.4 Calculation

$$\text{Benzene insolubles, percent by mass} = \frac{M_2 - M_1}{M} \times 100$$

where

M_2 = mass in g of the crucible along with insolubles,

M_1 = mass in g of the crucible, and

M = mass in g of the *prepared sample* taken for the test.

A-6. DETERMINATION OF MELTING POINT

A-6.1 Determine the melting point of the *prepared sample* (**A-1.1**) as prescribed in **8** of IS : 5299-1969*.

*Methods of sampling and tests for dye intermediates.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Conversion*
Force	newton	N	1 N = 1 kg.1 m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

PUBLICATIONS OF INDIAN STANDARDS INSTITUTION

INDIAN STANDARDS

Over 8 500 Indian Standards covering various subjects have been issued so far. Of these, the standards belonging to the Chemical Group fall under the following categories:

Acids
Adhesives
Alcohols and allied products
Alkalis
Brushware
Cermicware, enamelware and laboratory porcelain
Chemical hazards and safety
Chemicals, inorganic (miscellaneous)
Chemicals, organic (miscellaneous)
Coal and coke
Coal carbonization products
Coated fabrics
Cosmetics and toilet goods
Drying oils
Dye intermediates
Electroplating chemicals
Explosive and pyrotechnic materials
Fertilizers
Fillers, stoppers and putties
Footwear
Glass and glassware
Industrial gases
Inks and allied products
Laboratory glassware thermometers and related apparatus
Lac and lac products
Leather, leather goods and leather dressings
Linters and allied products
Lubricants and related products
Oil pastes
Oils & fats, oleaginous seeds and fruits
Painters' materials (miscellaneous)
Paper and its products
Paper and pulp board packaging materials
Perfumery materials, natural and synthetic
Petroleum and petroleum products
Photographic chemicals
Pigments and extenders
Plastics
Polishes
Printing inks
Ready mixed paints and enamels
Rubber and rubber products
Soaps and other surface active agents
Tanning materials and allied products
Thermal insulation materials
Thinners and solvents
Varnishes and lacquers
Water and water treatment
Water based paints
Unclassified

OTHER PUBLICATIONS

ISI Bulletin (Published Every Month)	
Single Copy	Rs 4·00
Annual Subscription	Rs 36·00
Standards : Monthly Additions	
Single Copy	Re 0·30
Annual Subscription	Rs 3·00
Annual Reports (from 1948-49 Onwards)	Rs 2·00 to 6·00
ISI Handbook, 1975	Rs 30·00

INDIAN STANDARDS INSTITUTION

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002
Telephone : 27 01 31 (20 lines) **Telegrams : Manaksanstha**

Regional Offices :		Telephone
Western : Novelty Chambers, Grant Road	BOMBAY 400007	37 97 29
Eastern : 5 Chowringhee Approach	CALCUTTA 700072	23-08 02
Southern : C.I.T. Campus, Adyar	MADRAS 600020	41 24 42
Branch Offices :		
'Pushpak', Nurmohamed Shaikh Marg, Khanpur	AHMADABAD 380001	2 03 91
'F' Block, Unity Bldg, Narasimharaja Square	BANGALORE 560002	2 76 49
Ahimsa Bldg, SCO 82-83, Sector 17C	CHANDIGARH 160017	2 83 20
5-8-56/57 L. N. Gupta Marg	HYDERABAD 500001	4 57 11
117/418 B Sarvodaya Nagar	KANPUR 208005	82 72
B.C.I. Bldg (3rd Floor), Gandhi Maidan East	PATNA 800004	5 36 55
Hantex Bldg (2nd Floor), Rly Station Road	TRIVANDRUM 695001	32 27

Printed at Delhi Printers, New Delhi, India